GENESIS SOLAR WIND SAMPLE 61422: EXPERIMENT IN VARIATION OF SEQUENCE OF CLEANING SOLVENT FOR REMOVING CARBON-BEARING CONTAMINATION. J. H. Allton[1] and K. R. Kuhlman[2], K. K. Allums[3], C. P. Gonzalez[3], A. J. G. Jurewicz[4], D. S. Burnett[5], and D. S. Woolum[6]. [1]NASA/Johnson Space Center, Mail Code XI, 2101 NASA Parkway, Houston, TX, 77058, judith.h.allton@nasa.gov, [2]Planetary Science Institute, Tucson, AZ, [3]Jacobs, NASA Johnson Space Center, Houston, TX, [4]Arizona State University, Tempe, AZ, [5]California Institute of Technology, Pasadena, CA, [6]California State University, Fullerton, CA.

Introduction The recovered Genesis collector fragments are heavily contaminated with crash-derived particulate debris. However, megasonic treatment with ultra-pure-water (UPW; resistivity >18 meg-ohm-cm) removes essentially all particulate contamination greater than 5 microns in size [e.g.1] and is thus of considerable importance. Optical imaging of Si sample 60336 revealed the presence of a large C-rich particle after UPW treatment that was not present prior to UPW[2]. Such handling contamination is occasionally observed, but such contaminants are normally easily *removed* by UPW cleaning. The 60336 particle was exceptional in that, surprisingly, it was not removed by additional UPW or by hot xylene or by aqua regia treatment [2]. It was eventually removed by treatment with NH_3-H_2O_2 [3]. Our best interpretation of the origin of the 60336 particle was that it was adhesive from the Post-It notes used to stabilize samples for transport from Utah after the hard landing [4]. It is possible that the insoluble nature of the 60336 particle comes from interaction of the Post-It adhesive with UPW. An occasional bit of Post-It adhesive is not a major concern, but C particulate contamination also occurs from the heat shield of the Sample Return Capsule (SRC) and this is mixed with inorganic contamination from the SRC and the Utah landing site. If UPW exposure also produced an insoluble residue from SRC C, this would be a major problem in chemical treatments to produce clean surfaces for analysis. This paper reports experiments to test whether particulate contamination was removed more easily if UPW treatment was not used.

A series of cleaning steps on silicon sample 61422, which was not transported on a Post-It note, began with organic solvent xylene instead of UPW.

Cleaning and Documentation Sequence: a) optical imaging; b) batch cleaning with hot xylene, ultrasonic in a Pyrex beaker; c) optical imaging; d) SEM particle analysis; e) optical imaging; f) UV ozone surface treatment; g) optical imaging; h) UPW megasonic flow; i) optical imaging. Special regions were marked for higher resolution optical imaging (Fig. 1).

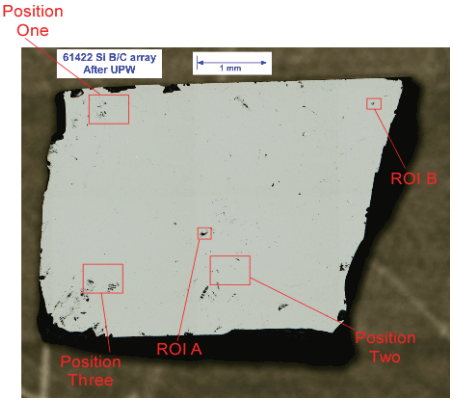

Fig. 1 Locations of features.

Effects of Xylene Batch Cleaning Followed by UV Ozone and UPW: Batch cleaning with xylene is not as effective as UPW spin cleaning at removing general particulates. Dried droplets of the final acetone rinse clearly show that crash debris has been redistributed. Small germanium particles serve nicely as markers of spacecraft debris. [Nineteen Ge solar wind collectors were reduced to powder during the crash, and these fines are ubiquitous.] The region shown in Fig. 2 illustrates the efficacy of UV ozone followed by UPW in reducing organics and particulates.

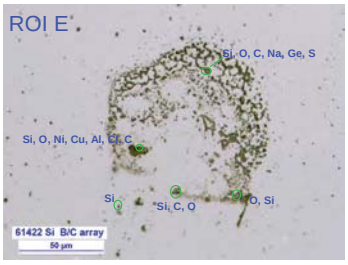

Fig. 2a. Dried droplet with particle composition determined by SEM. This feature was not observed prior to xylene cleaning.

Fig. 2b. Droplet from 2a after UV ozone (left) and after UPW (right).

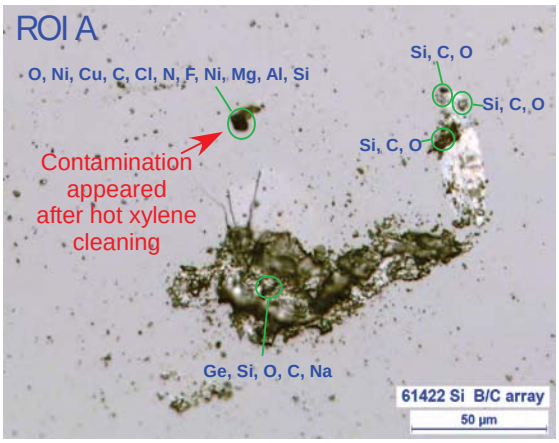

Fig. 3. Optical image of ROI A after xylene cleaning and SEM composition assessment.

Composition Maps in a Complex Feature: Examples from a complex C-rich particle, of "scorpion" shape and resulting from the crash, are observed in Region of Interest A (ROI A) (Fig. 3). EDX mapping shows small particles containing Ge, Ga, Mg, As, and Al in the feature of interest (Figs. 4 & 5). These appear to exist within a smudge or trough created in the Si. Mapping area shown in RED rectangle, Fig. 4. In addition to mapping, linear traverses were made for Si, C, O, and Ge. Germanium and oxygen peaks were more sharply defined than carbon.

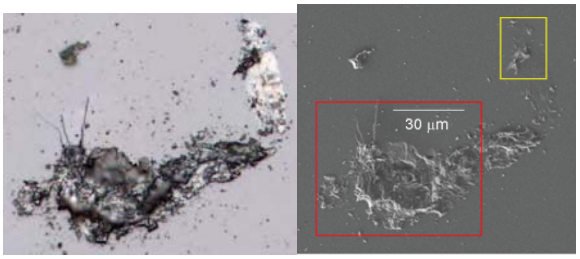

Fig. 4. ROI A, Left: optical Right: SEM.

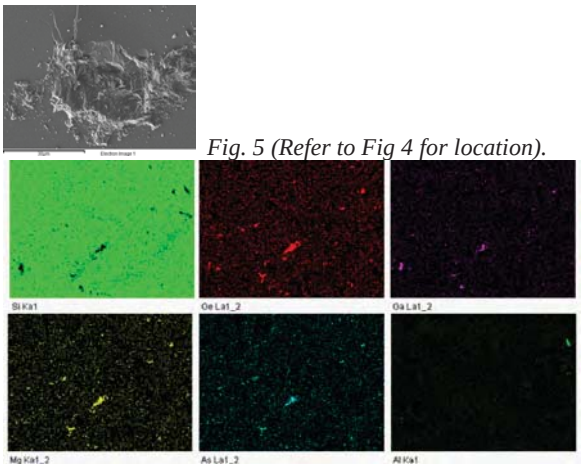

Fig. 5 (Refer to Fig 4 for location).

Effect of Cleaning on reduction of Carbon-Bearing Contaminants: An attempt was made to optically track changes in the area of specific carbon-bearing particles in the "scorpion tail" (area framed in yellow Fig. 4). Optical resolution was not adequate for comparable measurements, but it is evident that batch xylene ultrasonication removed a few loose small particles, but continuous flow UPW megasonic removed many more (Figs. 6a & 6b). In region of interest B (ROI B) 8 spectra were taken with particles identified: 5 silicon; 2 C, O, Si; and 1 Ge. Only the germanium was removed by UPW cleaning (Fig. 7).

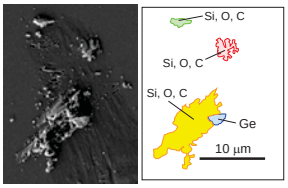

Fig. 6a. SEM. "Scorpion tail" carbon particle areas: green, 2.6 µm^2; pink, 6.1 µm^2; yellow, 45.3 µm^2.

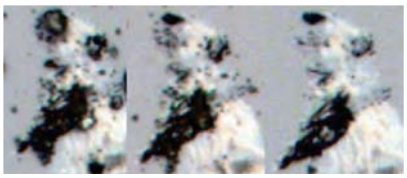

Fig. 6b. Optical. Cleaning sequence: original, post xylene, post UPW.

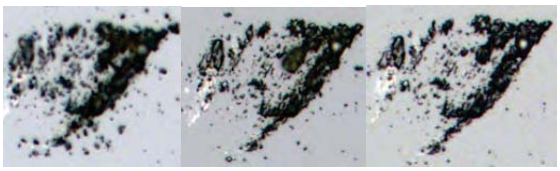

Fig. 7. ROI B cleaning sequence: original, post xylene, post UPW. FOV for each frame is 50 µm

Summary: The above results are typical or slightly worse than for samples initially cleaned with UPW. Thus there is no evidence that an initial UPW cleaning makes particulate removal more difficult as was possible based on the 60336 particle. Further, 1) UPW megasonic flow removes more particles than batch ultrasonic xylene; 2) UV ozone is effective at removing recently-dried organic film. This study supported previous studies indicating that silicon and germanium are ubiquitous, but that Ge is more easily moved than silicon by the cleaning protocols employed [5].

References: [1] Gonzalez C. P. *et al.* (2014) LPS 45th, abst. # 2127. [2] Goreva Y. S. *et al.* (2014) LPS 45th, abst. #2245. [3] Goreva Y. S. *et al.* (2015), 46th. [4] Allums K. K. *et al.* (2015), LPS 46th, . [5] Schmeling M. *et al.* (2011), LPS 42nd, abst. #2041.